

Low-temperature demagnetization of saturation remanence in magnetite-bearing dolerites of high coercivity

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Accepted 1997 September 12. Received 1997 August 7; in original form 1997 January 24

SUMMARY

We studied 16 magnetite-bearing dolerite dyke samples of high coercive force (H_C ranging from 11 to 50 mT) that had been used successfully in Precambrian palaeomagnetic studies. Each dolerite was given a saturation remanent magnetization, whose change was measured as the sample was cooled to 77 K in zero field and warmed back to room temperature. Only the three dolerites of highest H_C (≥ 40 mT) show little change on cooling, suggesting that their magnetite is mostly in elongated single-domain grains. The rest of the dolerites are likely to be dominated by pseudo-single-domain magnetite. Cooling to ~ 135 K causes their remanence to decrease (by 37 per cent on average) in rough proportion to the decrease in saturation magnetostriction, as expected if internal stresses oppose domain wall motion. Cooling from ~ 135 K to 77 K causes remanence to decrease further (by 26 per cent on average), probably mostly because of domain reorganization forced by magnetite's Verwey crystallographic transition. Warming back to room temperature causes some of the remanence loss to be recovered, perhaps because internal stresses act as a bridge between different easy axes below the Verwey temperature (~ 122 K) and above the isotropic point (~ 135 K). This recoverable low-temperature demagnetization averages 23 ± 6 per cent of the initial saturation remanence, while the permanent demagnetization averages 40 ± 9 per cent. Recoverable low-temperature demagnetization is even larger for natural remanence, averaging 46 ± 9 per cent for the six dolerites measured, while the corresponding permanent demagnetization averages 13 ± 6 per cent. Large recoverable low-temperature demagnetization seems to be characteristic of pseudo-single-domain magnetite in which high internal stresses block domain-wall motion, and may be common in mafic igneous rocks like our dolerite samples, whose magnetite is intergrown with ilmenite lamellae. Measuring natural remanence of such rocks before, after and while at 77 K should help separate remanence carried by multidomain magnetite (mostly permanently demagnetized), by single-domain magnetite (mostly unchanged) and by pseudo-single-domain magnetite (mostly responsible for recoverable demagnetization).

Key words: demagnetization, magnetic domain, magnetite, palaeomagnetism, rock magnetism.

1 INTRODUCTION

Magnetite grains subdivided into fine particles by ilmenite lamellae are common in mafic igneous rocks and are important recorders of the Earth's magnetic field (Larson *et al.* 1969). Recently, such magnetite in dolerite dykes has become very important to Precambrian palaeomagnetism, because often the

dykes are stably magnetized and yield precise U–Pb dates (Buchan & Halls 1990). In this paper, we study mechanisms of low-temperature demagnetization of saturation remanence in Precambrian dolerite dyke samples bearing magnetite with ilmenite lamellae. This helps test the suggestion of Hodych (1996) that these dolerites often owe the high coercivity of their remanence to pseudo-single-domain magnetite in which internal stresses block domain wall motion. We also investigate whether low-temperature demagnetization can help separate remanence carried by multidomain, pseudo-single-domain and

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single-domain magnetite in such rocks, extending the work of Levi & Merrill (1978), Dunlop & Argyle (1991), Heider, Dunlop & Soffel (1992) and Halgedahl & Jarrard (1995).

Our dolerite samples are given a saturation remanent magnetization (J_{RS}) at room temperature. Then they are placed in a field-free space where magnetization changes are measured as the samples are cooled to liquid-nitrogen temperature (77 K) and warmed back to room temperature. This cycle to 77 K usually causes some permanent demagnetization of the samples. Three main mechanisms for such low-temperature demagnetization have been suggested for magnetite containing domain walls. First, as suggested by Ozima, Ozima & Akimoto (1964), demagnetization may be caused by magnetite's magneto-crystalline anisotropy constant K_1 decreasing on cooling (becoming zero at magnetite's 'isotropic' point, $T_K \approx 135$ K) and unblocking domain walls. This mechanism may be important when magnetite's internal stresses are low, as is likely for the large magnetite crystals studied by Ozima *et al.* (1964), Kobayashi & Fuller (1968) and Halgedahl & Jarrard (1995). However, a second mechanism may dominate when internal stresses are high (as in magnetite subdivided by ilmenite lamellae): demagnetization may then be caused by saturation magnetostriction decreasing on cooling, unblocking domain walls whose motion is opposed by internal stresses [or allowing domain walls to nucleate, as suggested by Halgedahl & Jarrard (1995)]. Hodych (1991) showed that this second mechanism is likely to dominate over the first in four basalts with coercive force between 6 and 13 mT, since their saturation remanence decreases in rough proportion to saturation magnetostriction rather than to K_1 on cooling to ~ 135 K in zero field. The importance of internal stresses to low-temperature demagnetization was also shown by the experiments of Heider *et al.* (1992), in which the internal stresses of synthetic magnetites were varied by quenching and annealing. A third mechanism for low-temperature demagnetization (Hodych 1991; Halgedahl & Jarrard 1995) is domain reorganization forced by cooling through magnetite's Verwey crystallographic transition (at $T_V \approx 122$ K).

Our dolerite samples range to much higher coercive force (50 mT) than the basalts studied by Hodych (1991). We find that saturation remanence in most of our samples decreases in approximate proportion to saturation magnetostriction on cooling to ~ 135 K, as expected if coercivity is due to internal stresses opposing domain wall motion. Only the three dolerite samples of highest coercive force (≥ 40 mT) show little change on cooling to ~ 135 K, as expected of elongated single-domain grains. We also test to what extent the Verwey transition is involved in low-temperature demagnetization.

The saturation remanence loss observed while cooling to 77 K is usually partly recovered on warming back to room temperature in zero field. This is perhaps due to the magnetite's internal stresses (Kobayashi & Fuller 1968; Heider *et al.* 1992). This recoverable low-temperature demagnetization seems characteristic of the pseudo-single-domain magnetite with high internal stresses in our dolerite samples and is even larger for their natural remanence than for saturation remanence. This novel finding supports the importance of internal stresses in pseudo-single-domain magnetite as a source of natural-remanence stability in mafic igneous rocks. It also suggests that measuring recoverable low-temperature demagnetization may be useful to palaeomagnetists in isolating the stable

natural remanence component carried by pseudo-single-domain magnetite with high internal stresses.

2 MAGNETIC PROPERTIES OF THE DOLERITES

The sixteen dolerites studied (Table 1) are all from separate Precambrian dykes. Most samples are from an east-west-trending dyke swarm near Nain, Labrador (Wiebe 1985). The exceptions (BX86, SD78 and RE88) are from the Biscotasing, Matachewan and Mackenzie dyke swarms respectively. Apart from a small soft overprint, all samples carry a stable natural remanent magnetization that is likely to be primary (Buchan & Halls 1990; Buchan *et al.* 1996). The alternating field required to demagnetize the remanence to half its initial value ($H_{1/2}$ in Table 1) is, on average, only 25 per cent lower for saturation remanence than for natural remanence.

The magnetic mineralogy and magnetic properties of our dolerite samples were studied by Hodych (1996), whose findings are summarized as follows. Most of our dolerite samples contain magnetite grains subdivided into fine particles by ilmenite lamellae. These magnetite particles are almost free of titanium, as shown by the Curie temperatures of our samples, which all lie between 580 and 560 °C. The magnetite is not significantly oxidized in most of our samples, judging by sharp Verwey transitions (Özdemir, Dunlop & Moskowitz 1993) in all but the three dolerites of highest coercive force ($H_C \geq 40$ mT). These three dolerites (samples 9144, 4305 and 5901) show little change in H_C on cooling, suggesting that they are dominated by single-domain magnetite grains with shape anisotropy. The other 13 dolerites are likely to be dominated by pseudo-single-domain magnetite, as shown by J_{RS}/J_S (the ratio of saturation remanence to saturation magnetization), which ranges from 0.10 to 0.32 and increases in approximate proportion to H_C in the suite of samples. Coercive force is likely to be dominantly controlled through internal stresses opposing domain wall motion, judging by the magnitude of the decrease in H_C on cooling to ~ 135 K. Magnetic interaction between the magnetite particles separated by ilmenite lamellae can be neglected to a first approximation, as discussed in detail by Hodych (1996).

Fine magnetite grains exsolved from silicate minerals may also be a significant source of high coercivity in some dolerites (Zhang & Halls 1995). However, our samples do not have the clouded feldspars characteristic of such dolerites. If magnetite grains exsolved from silicates are present, they should have high internal stresses and should thus behave like the grains of magnetite with exsolved ilmenite lamellae that dominate in our dolerite samples.

3 METHOD AND RESULTS OF THE LOW-TEMPERATURE DEMAGNETIZATION EXPERIMENTS

3.1 Saturation remanence given at room temperature

Each 1–2 cm³ dolerite sample was saturated at room temperature in a 350 mT direct field (after three-axis tumble demagnetization in a 350 mT alternating field). The sample was then placed in a field-free space where it was cooled in steps to liquid-nitrogen temperature and warmed in steps back to room temperature. Remanence intensity was measured after

Table 1. Magnetic properties of our dolerite samples (and of 210–250 μm magnetite grains in silicone cement). H_C is the coercive force; J_{RS}/J_S is the ratio of saturation remanence to saturation magnetization; $H_{1/2}$ is the alternating field strength required to reduce the saturation remanence (J_{RS}) or natural remanence (NRM) to half its initial value; ‘% Permanent’ and ‘% Recoverable’ low-temperature demagnetization are respectively the percentage of initial remanence that is permanently demagnetized on cooling to 77 K in zero field and the percentage that is recovered on warming back to room temperature in zero field.

Sample	H_C (mT)	J_{RS}/J_S	low-temperature demagnetization					
			$H_{1/2}$		% Permanent		% Recoverable	
			J_{RS} (mT)	NRM (mT)	J_{RS}	NRM	J_{RS}	NRM
9116	11.0	0.10	10.1	16.3	50 ± 1		34	
BX86	12.9	0.13	12.1	13.0	49 ± 2		22	
SD78	15.2	0.18	12.6	16.2	51 ± 1		22	
RE88	15.9	0.16	15.8	19.3	39 ± 4		31	
3301	18.2	0.17	14.1	17.4	34 ± 1	7	26	44
9138	22.0	0.20	18.4	25.0	32 ± 1		14	
3203	22.3	0.22	17.3	21.0	49 ± 1	16	27	56
2701	27.4	0.29	20.2	32.4	44 ± 2	6	24	55
3101	28.3	0.28	22.7	29.8	40 ± 3	17	24	47
4602	28.8	0.28	23.6	38.4	38 ± 2	22	22	40
5601	31.9	0.29	28.8	44.6	40 ± 2	12	20	31
9102	36.8	0.35	30.2	46.2	17 ± 1		13	
9128	38.2	0.32	31.9	45.0	36 ± 1		18	
5901	39.6	0.37	35.2	41.0	17 ± 3		6	
4305	40.9	0.42	38.6	46.2	9 ± 2		3	
9144	50.4	0.45	55.4	61.5	-7 ± 7		-5	
250–210 μ magnetite	2.7	0.02	8.2		78 ± 4		7	

each step when thermal equilibrium had been attained (which required about 25 minutes).

The apparatus used for the low-temperature demagnetization experiments is described in detail by Mackay (1995). Field-free space is provided by a set of five nested, high-permeability cylindrical cans. The sample is placed inside a non-inductively wound chromel A heating coil which is surrounded by a partially evacuated Dewar flask immersed in a liquid-nitrogen bath. The sample's temperature can be varied from room temperature to ~ 100 K by adjusting the alternating current through the heating coil, and 77 K can be attained by pouring liquid nitrogen into the sample holder. Magnetization is measured with a fluxgate probe (Schonstedt Model PSM-1) placed near the sample, inside the field-free space but outside the liquid-nitrogen bath, insulated from temperature change. The error in temperature measurement is usually about ± 4 K (greater near room temperature, owing to larger thermal gradients). The error in magnetization measurements is about ± 3 per cent of the initial room-temperature remanence intensity.

The solid circles joined by solid lines in Figs 1–3 plot remanence intensity (as a fraction of its initial room-temperature value) versus temperature for a representative suite of the samples. These curves are usually reproducible to within the errors of measurement quoted above, provided we wait ~ 25 minutes per temperature step to attain thermal equilibrium. If we wait about half this time, the cooling curves are shifted by ~ 15 K towards lower temperature, probably because the temperature of the sample lags behind that of the thermocouple. [Such thermal lag seems to have affected the low-temperature demagnetization curves of Hodych (1991),

which were done more quickly than in the present study and are displaced to lower temperature, judging by sample SD78.]

In some cases, the sample was remagnetized in 350 mT at room temperature and cooled to only ~ 135 K before warming back to room temperature. The warming curves are shown by open circles joined by solid lines in Figs 1 and 2.

3.2 Saturation remanence given at 77 K

After three-axis tumble demagnetization in a 350 mT alternating field, some of the samples were cooled to 77 K and then magnetized in a 350 mT direct field. These samples were then warmed in steps in zero field and the remanence was measured at each step (after waiting ~ 25 minutes for thermal equilibrium). The apparatus and errors of measurement were the same as in Section 3.1. The resulting data points are plotted as stars joined by dotted lines in Figs 1–3.

3.3 Natural remanence

The same apparatus as for saturation remanence was used to measure the change in intensity of natural remanence on cooling to ~ 100 K and warming to room temperature in zero field for six of the dolerite samples dominated by pseudo-single-domain magnetite. Only the remanence intensity along the initial remanence direction was measured since little change in direction was observed during alternating-field demagnetization of these samples. The permanent and recoverable low-temperature demagnetizations on cooling to ~ 100 K are

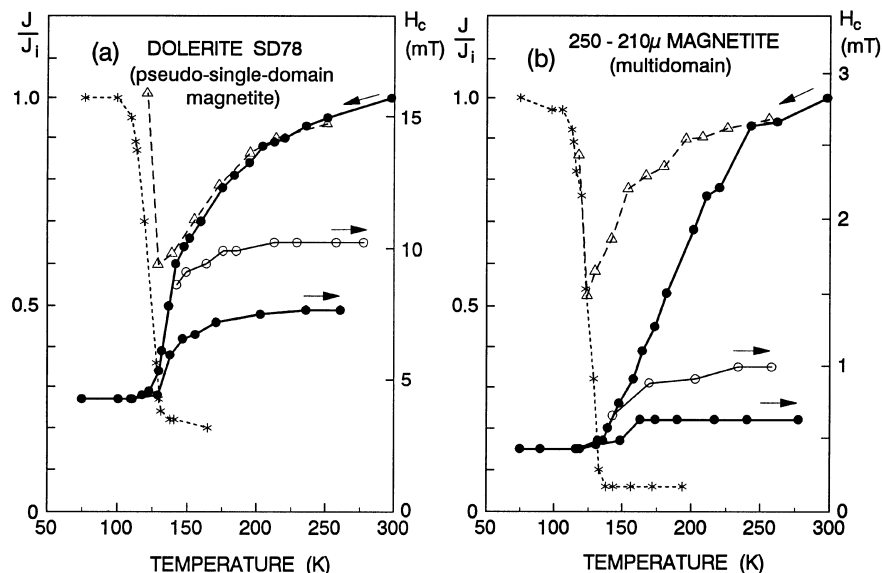


Figure 1. Comparison of (a) SD78, typical of our dolerite samples dominated by pseudo-single-domain magnetite, with (b) 210–250 μm crushed multidomain magnetite. Solid circles joined by solid lines show saturation remanence (as a fraction J/J_i of its initial room-temperature value) as it changes in zero field on cooling to 77 K and warming back to room temperature. Open circles joined by solid lines show the effect of warming after cooling to only ~ 135 K. Stars joined by dotted lines show change in saturation remanence given at 77 K and then warmed in zero field. Open triangles joined by dashed lines show change in coercive force (H_c) on cooling to ~ 100 K.

listed as a percentage of the initial room-temperature natural remanence in Table 1.

3.4 Coercive force

For each 1–2 cm^3 dolerite sample, we measured the change in coercive force H_c on cooling to ~ 100 K using apparatus described in detail by English (1995). A vibrating-sample magnetometer was used to trace hysteresis loops as the field was cycled to ~ 350 mT with an electromagnet. A similar temperature-control method was used to that in low-temperature demagnetization. The error in temperature measurement was usually about ± 3 K (greater near room temperature). The error in H_c measurement was usually about ± 0.4 mT. Coercive force data are shown by open triangles joined by dashed lines in Figs 1–3.

4 DISCUSSION OF DOLERITES DOMINATED BY PSEUDO-SINGLE-DOMAIN MAGNETITE

13 of our 16 dolerite samples are likely to be dominated by pseudo-single-domain magnetite (Hodych 1996) and will now be discussed together. Representative results from five of these 13 dolerites are illustrated in Figs 1(a) and 2. For each dolerite, cooling to 77 K in zero field causes the saturation remanence to decrease. Part of this decrease is permanent and part recovers on warming back to room temperature in zero field. The permanent part of the low-temperature demagnetization and the recoverable part (given by the remanence increase on warming from 77 K back to room temperature) are listed as a percentage of the initial room-temperature remanence in Table 1. On average, the five dolerites of Figs 1(a) and 2 show 45 ± 7 per cent permanent demagnetization and 24 ± 7 per cent recoverable demagnetization on cooling to 77 K, in agreement with the 37 ± 10 per cent permanent and 22 ± 6 per cent

recoverable demagnetization shown on average by the other eight dolerites.

4.1 Saturation remanence on cooling to ~ 135 K

Cooling to ~ 135 K causes H_c to decrease, which should unpin some of the domain walls, allowing self-demagnetizing fields to reduce the remanence. Even at room temperature, self-demagnetizing fields shear hysteresis loops in multidomain magnetite and should (Néel 1955) result in

$$J_{RS} \approx 10^{-3} H_c / N \quad (J_{RS} \approx H_c / N \text{ in cgs}). \quad (1)$$

Here, N is the average self-demagnetizing factor of the magnetite grains and SI units are used, except that H_c is expressed not in A m^{-1} but in the millitesla equivalent. Hence, on cooling to ~ 135 K in zero field, provided H_c decreases monotonically, we expect the saturation remanence in multidomain magnetite to decrease in approximate proportion to H_c . The same should be true of pseudo-single-domain magnetite if its coercivity is due to opposition to domain wall motion. Vortex structures (Williams & Dunlop 1995) can probably be neglected because of the high coercivity of the pseudo-single-domain magnetite in our dolerite samples (Hodych 1996).

If domain wall motion is opposed by internal stresses, theory [Néel (1946), to first approximation] predicts

$$H_c \propto \sigma \lambda_s / J_s. \quad (2)$$

The internal stresses σ may be those associated with dislocations (Xu & Merrill 1992; Moskowitz 1993) and will be considered invariant compared to the saturation magnetostriction λ_s on cooling to ~ 135 K [since σ should vary in approximate proportion to the shear modulus (Träuble 1969), which changes by < 8 per cent between 500 and 300 K (Bhagavantam 1955)]. Combining eqs (1) and (2) gives

$$J_{RS} \propto \sigma \lambda_s / J_s N. \quad (3)$$

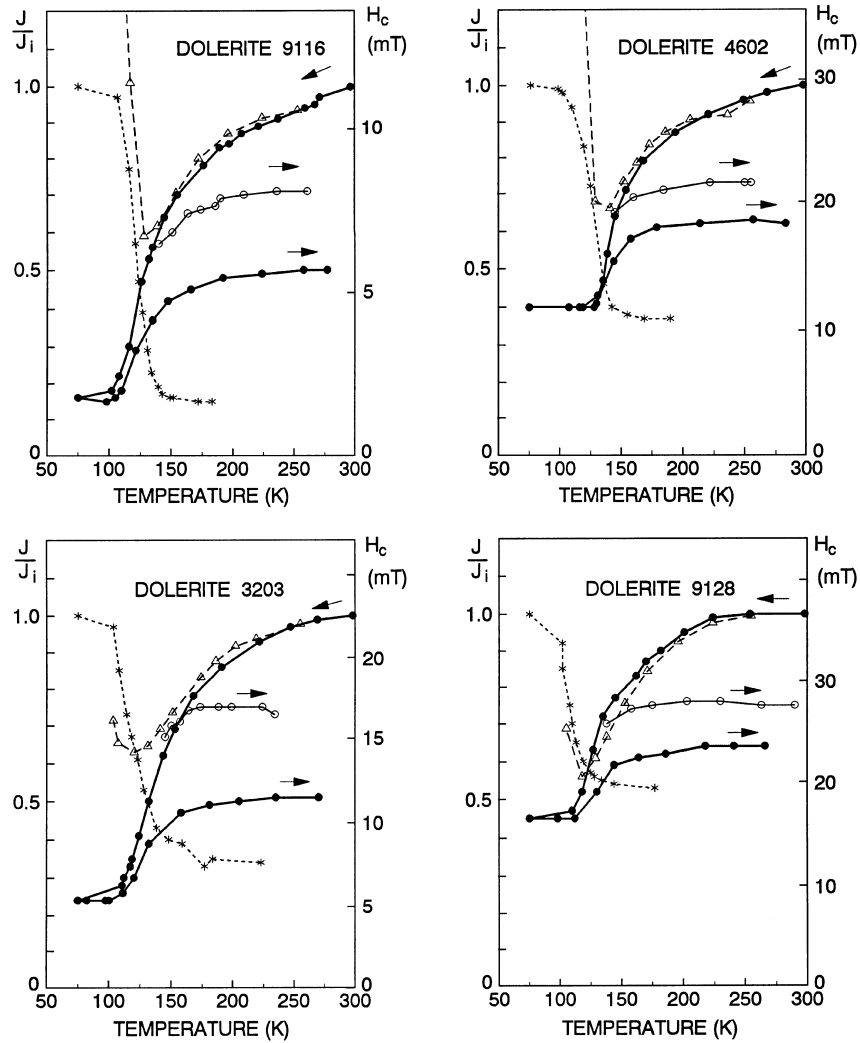


Figure 2. Typical low-temperature behaviour of the dolerite samples that are dominated by pseudo-single-domain magnetite. Symbols are as in Fig. 1.

That is, we expect the saturation remanence to decrease in approximate proportion to λ_s/J_s on cooling to ~ 135 K if domain wall motion is opposed by internal stresses in multidomain or pseudo-single-domain magnetite. Since λ_s for magnetite is not isotropic (as assumed by theory), we substitute $\bar{\lambda}_s = 0.4\lambda_{100} + 0.6\lambda_{111}$, the saturation magnetostriction expected for polycrystalline magnetite.

Cooling to ~ 135 K does cause a monotonic decrease in H_c , and J_{RS} does decrease in approximate proportion to H_c , as expected from eq. (1), in all of our dolerites dominated by pseudo-single-domain magnetite [and in the four basalts of Hodych (1991)]. This can be seen in Figs 1(a) and 2, where the curves of remanence intensity versus temperature are all of very similar shape to the curves of H_c versus temperature on cooling to ~ 135 K. It can also be seen in Fig. 4(a), where the remanence is plotted as a function of H_c as both of these decrease on cooling to ~ 135 K. All the plots for the pseudo-single-domain dolerites (closed symbols) are linear and, when extrapolated, many pass close to the origin, as expected from eq. (1). A few plots pass well above or below the origin, perhaps because a small fraction of the magnetite is single-domain or multidomain respectively. According to eq. (1), the

slope of each line in Fig. 4(a) gives an estimate of the average N for the magnetite particles intergrown with ilmenite in the dolerite samples. These N values average 0.16 ± 0.03 (2.0 ± 0.4 in cgs). This agrees with the average $N \sim 0.17$ (2.1 in cgs) estimated from the slope of the room-temperature H_c versus J_{RS}/J_s plot for the suite of dolerites (Hodych 1996).

Since H_c for many of these dolerites has been shown to decrease in approximate proportion to $\bar{\lambda}_s/J_s$ on cooling to ~ 135 K (English 1995), we expect the saturation remanence to decrease in approximate proportion to $\bar{\lambda}_s/J_s$. The plots (Fig. 4b) for the pseudo-single-domain dolerites (closed symbols) are linear and pass close to the origin when extrapolated, as expected from eq. (3) if remanence coercivity is due to internal stresses opposing domain wall motion. The plot for dolerite 9102 passes well above the origin, perhaps because a significant fraction of its magnetite is single-domain. [The low-temperature variations of $\bar{\lambda}_s$ and J_s are from the measurements of Bickford, Pappis & Stull (1955) and Pauthenet (1950) respectively and J_0 is the value of J_s at absolute zero.]

In contrast, for low-coercivity multidomain magnetite, the saturation remanence decreases much faster than H_c on cooling, as can be seen in Fig. 1(b) for the sample of 210–250 μm

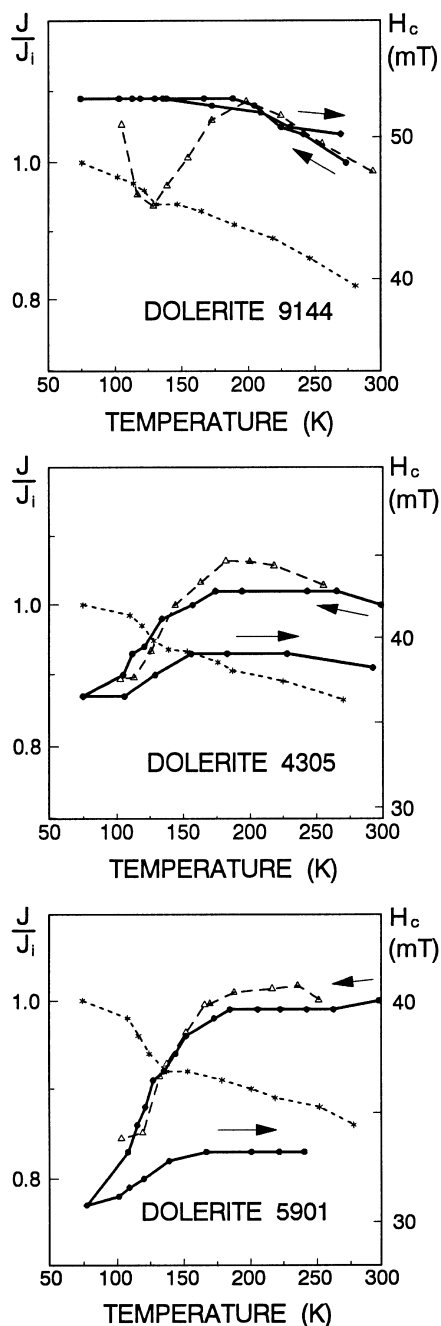


Figure 3. Low-temperature behaviour of our three dolerite samples of highest coercive force, which are likely to be dominated by elongated single-domain magnetite particles. Symbols are as in Fig. 1.

crushed natural magnetite dispersed in silicone cement. [The H_C data are from Hodych (1986).] This is also true for the two mafic rocks of Hodych (1991) with lowest H_C (1.1 and 2.1 mT) and lowest J_{RS}/J_S (0.02). Perhaps barriers to domain wall motion are so low that cooling demagnetizes not only by lowering coercivity but also by allowing local reorganization of domain structure. Such reorganization may have been responsible for some of the large Barkhausen jumps in remanence observed by Halgedahl & Jarrard (1995) while cooling a large single crystal of magnetite.

In our 13 dolerites, cooling to ~ 135 K in zero field causes the saturation remanence to decrease by 37 ± 10 per cent on

average. This is close to the ~ 33 per cent decrease in \bar{J}_S/J_S on cooling to 135 K, as expected from eq. (3). In the five dolerites of Figs 1(a) and 2, cooling to ~ 135 K causes a 39 ± 6 per cent average decrease in saturation remanence, consisting of a 29 ± 4 per cent decrease that is permanent and a 10 ± 4 per cent decrease that is recovered on warming the dolerite back to room temperature in zero field. (The remanence change while warming is shown by open circles in Figs 1a and 2).

4.2 Cooling from ~ 135 K to 77 K

On cooling through magnetite's isotropic point at ~ 135 K [± 6 K, averaging T_K from Syono (1965) and Bickford, Brownlow & Penoyer (1957)], the cubic magnetocrystalline anisotropy constant K_1 drops to zero and the easy axes switch from $[111]$ to $[100]$ directions. The magnetocrystalline anisotropy increases on further cooling but remains low until magnetite's Verwey transition at ~ 122 K (Chikazumi 1976), below which it increases greatly (Williams, Bozorth & Goertz 1953), acquiring a single easy axis along one of the former $[100]$ directions. Changes at both the isotropic point and the Verwey transition could force domain reorganization and remanence decrease on cooling. However, in our dolerites with their highly stressed magnetites, changes at the isotropic point should be relatively small, because (judging by the variation of H_C) stress-induced anisotropy dominates over the weak magnetocrystalline anisotropy.

The Verwey transition temperature T_V can be estimated by assuming that it approximately coincides with the temperature at which H_C is a minimum. Although the temperature of minimum H_C was not measured precisely in most of our dolerites, its average is 125 ± 4 K for the 13 dolerites and the 210–250 μm magnetite, which agrees with the $T_V = 122$ K (varying by ± 3 K depending on internal stresses) reported by Chikazumi (1976) for pure magnetite. A second method of estimating T_V is to give the dolerite a remanence at 77 K in a large field (we used 350 mT) and then to warm the dolerite in zero field. There should be a sharp decrease of remanence when T_V is reached, due to a large decrease in magnetocrystalline anisotropy. Such demagnetization curves are shown with stars joined by dotted lines for the six samples in Figs 1 and 2. On average, rapid remanence decrease is seen to begin at 108 ± 4 K and to end at 135 ± 4 K. Assuming that T_V is midway between these two values gives an average T_V of ~ 121 K, which is in good agreement with the 125 ± 5 K average temperature of minimum H_C in these six samples and with the $T_V \sim 122$ K quoted by Chikazumi (1976). The effects of the Verwey transition on remanence appear to extend over a 27 K interval, which may partly be due to variations in titanium content and internal stresses within the magnetite of each sample.

On cooling from 135 to 77 K, the saturation remanence in our 13 dolerites decreases by an additional 26 ± 9 per cent on average, with almost all this decrease complete by 111 ± 9 K. Most of the remanence decrease between 135 K and 111 K is likely to be associated not with an H_C decrease (which ceases at 125 ± 4 K) but with domain reorganization forced by the Verwey transition. This is supported by our observation above, that the saturation remanence given at 77 K demagnetizes on warming between ~ 108 K and 135 K.

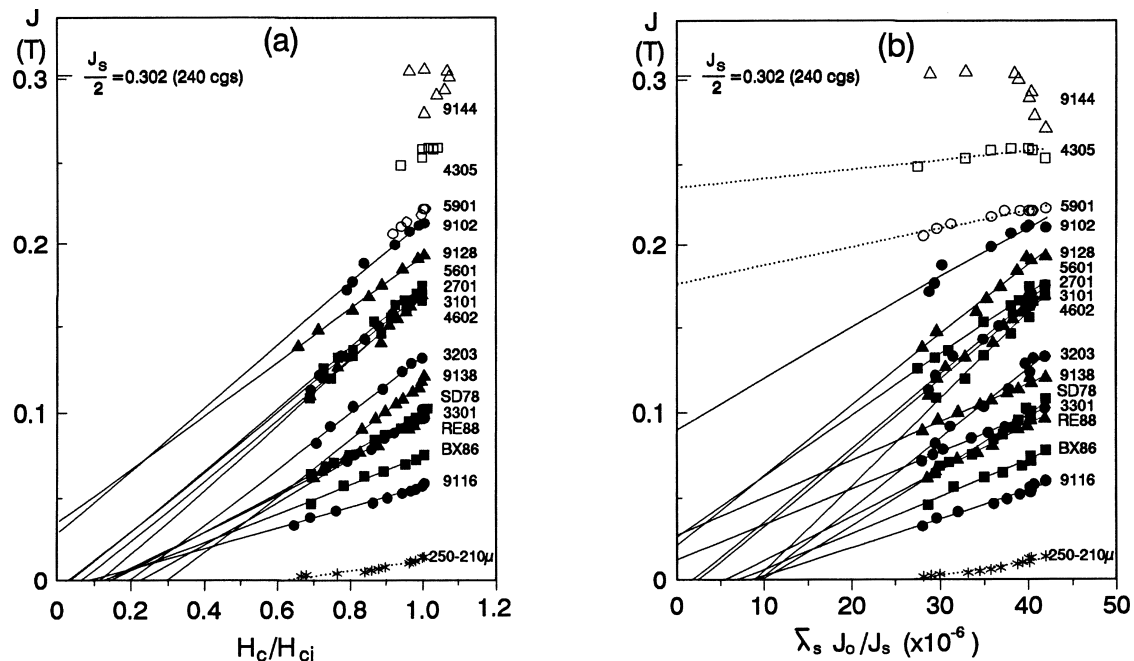


Figure 4. (a) Saturation remanence intensity J (per unit volume of magnetite in the sample) plotted as a function of coercive force (as a fraction H_c/H_{ci} of its value near room temperature) as our samples are cooled to ~ 135 K in zero field. Solid symbols are for dolerites whose magnetite is mostly pseudo-single-domain. Open symbols are for dolerites whose magnetite is likely to be single-domain. Stars are for 210–250 μ m crushed magnetite. (b) Saturation remanence intensity J plotted as a function of polycrystalline saturation magnetostriction $\bar{\lambda}_s$ divided by J_s/J_0 (the ratio of saturation magnetization to saturation magnetization at absolute zero) as our samples are cooled to ~ 135 K in zero field.

4.3 Warming back to room temperature from 77 K

Warming back to room temperature in zero field causes the remanence to increase. On average, for our 13 dolerite samples, 13 ± 5 per cent of initial room-temperature saturation remanence is recovered between ~ 111 K and ~ 135 K, and 10 ± 4 per cent is recovered between ~ 135 K and room temperature. (The latter agrees well with the 10 ± 4 per cent average remanence recovered in a cooling cycle to ~ 135 K for the five dolerites of Figs 1a and 2.)

The mechanism of this remanence recovery may be the following, described by Heider *et al.* (1992), who simplify a qualitative model proposed by Kobayashi & Fuller (1968). Upon cooling to the isotropic point at ~ 135 K, the magnetization in a given domain switches from one of four $[111]$ easy axes to the nearest local stress-induced easy axis. Upon further cooling through the Verwey transition at ~ 122 K, the magnetization switches from the stress-induced easy axis to the single magnetocrystalline easy axis. This sequence is reversed during heating, and parts of the magnetite with stronger stress-induced anisotropy have a better chance of recovering their original domain magnetization directions. This is because the stress-induced easy axis provides a bridge between the single magnetocrystalline easy axis below the Verwey transition and the originally occupied one of four $[111]$ axes above the isotropic point. The model is plausible since the likelihood of internal stress control of coercivity has been independently demonstrated, for example by H_c decreasing in approximate proportion to $\bar{\lambda}_s/J_s$ on cooling to ~ 135 K in many of our dolerite samples (English 1995).

Relatively high recoverable low-temperature demagnetization seems to be characteristic of remanence carried by pseudo-single-domain magnetite with high internal stress, judging by

our 13 dolerite samples with $H_c < 40$ mT and $J_{RS}/J_s \leq 0.35$ (Table 1). In contrast, low-coercivity multidomain magnetite is characterized by large permanent low-temperature demagnetization, and elongated single-domain magnetite grains should show little permanent or recoverable low-temperature demagnetization.

4.4 Comparison with the results of other workers on synthetic samples

Hartstra (1982) and Halgedahl & Jarrard (1995) published curves of how saturation remanence changes during cooling cycles to 77 K for synthetic samples containing pseudo-single-domain magnetite. Fig. 5(a) compares their results with ours for the two dolerites that most resemble their samples in room temperature J_{RS}/J_s and H_c . Our dolerite SD78 ($J_{RS}/J_s = 0.18$, $H_c = 15$ mT) and Hartstra's $< 5 \mu$ m magnetite ($J_{RS}/J_s = 0.17$, $H_c = 14$ mT) show almost the same ~ 72 per cent decrease in saturation remanence on cooling to 77 K. However, on cooling to ~ 135 K, dolerite SD78 shows a 45 per cent decrease whereas Hartstra's magnetite shows a 64 per cent decrease; the former is more consistent with the ~ 33 per cent decrease expected from eq. (3). Our dolerite BX86 and the 1.5μ m magnetite-in-glass-ceramic sample studied by Halgedahl & Jarrard (1995) have almost the same J_{RS}/J_s (0.13 and 0.14 respectively) and almost the same ~ 72 per cent decrease in saturation remanence on cooling to 77 K. However, on cooling to ~ 135 K, dolerite BX86 shows ~ 40 per cent decrease, whereas the 1.5μ m magnetite shows only ~ 17 per cent decrease. The former is more consistent with the ~ 33 per cent decrease expected from eq. (3).

Hartstra (1982) and Halgedahl & Jarrard (1995) also published curves of saturation remanence change during

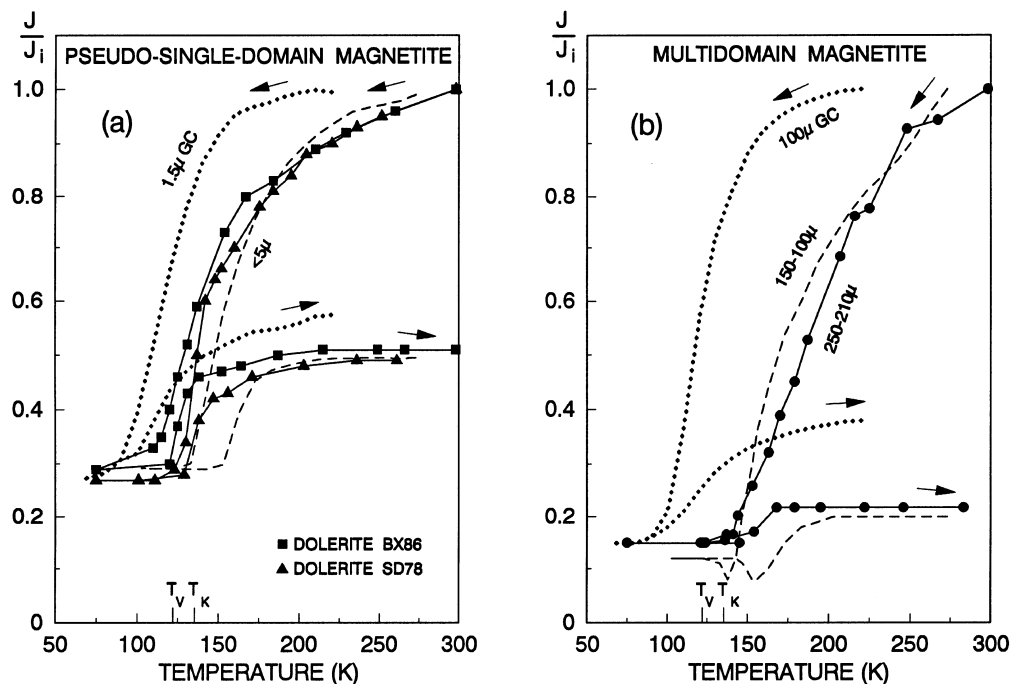


Figure 5. Comparison of our low-temperature demagnetization experiments (solid symbols joined by solid lines) with those of Halgedahl & Jarrard (1995) using glass-ceramic samples (dotted lines) and with those of Hartstra (1982) using samples with crushed magnetite (dashed lines). Saturation remanence (as a fraction J/J_i of its initial value near room temperature) is shown as it changes in a cooling cycle to 77 K in zero field. Samples in (a) are dominated by pseudo-single-domain magnetite and samples in (b) by multidomain magnetite. The Verwey transition temperature (T_V) and the isotropic point (T_K) for pure magnetite are indicated.

cooling cycles to 77 K for synthetic samples containing large, multidomain magnetite grains with $J_{RS}/J_S = 0.02$, as in our sample containing 210–250 μm crushed magnetite grains. As seen in Fig. 5(b), our sample and Hartstra's 100–150 μm crushed magnetite sample behave similarly on cooling, but differ from Halgedahl & Jarrard's glass-ceramic sample with 100 μm magnetite grains. Although the saturation remanence decreases by ~ 85 per cent in all three samples on cooling to 77 K, all of this decrease has occurred when 135 K is reached in our sample and that of Hartstra, whereas there is only a 22 per cent decrease at 135 K in the glass-ceramic sample. This 22 per cent decrease is unexpectedly small since the large grain size and low J_{RS}/J_S suggest low-coercivity multidomain magnetite grains, whose saturation remanence should decrease by more than the 33 per cent predicted by eq. (3).

Hodych (1996) pointed out that the pseudo-single-domain magnetite in glass-ceramic samples studied by Worm & Markert (1987) closely resembled our dolerites in range of and proportionality between J_{RS}/J_S and H_C values. This suggested that the highly stressed magnetite in the glass-ceramic samples might be a reasonable synthetic analogue for the magnetite in mafic igneous rocks. However, they may not be as good an analogue as samples containing crushed magnetite grains, judging by the decrease in saturation remanence observed during cooling to 77 K. We recommend testing this further with cooling experiments using the multidomain and pseudo-single-domain magnetite-in-glass-ceramic samples studied by Worm & Markert (1987). It is particularly important to test for internal stress control of coercivity by measuring whether H_C in these glass-ceramic samples varies in approximate proportion to the saturation magnetostriction on cooling as predicted by eq. (2). This seems to be true for their samples

bearing titanomagnetite with 0.55 or 0.17 mole fraction of ulvöspinel (Worm and Markert 1987) but has not been tested for their samples bearing pure magnetite.

4.5 Natural remanence

The natural remanence of six of our dolerite samples (3301, 3203, 2701, 3101, 4602 and 5601) that are dominated by pseudo-single-domain magnetite was subjected to low-temperature demagnetization. The natural remanence should be mainly a thermal remanence acquired when the dolerites originally cooled in the Precambrian. Although a viscous component is also present, it makes up only 8 per cent or less of the natural remanence, and can be removed by 15 mT or less of alternating-field demagnetization.

The average permanent low-temperature demagnetization is 13 ± 6 per cent for natural remanence, which is much smaller than the 41 ± 5 per cent average for saturation remanence in these six samples. The smaller permanent demagnetization is likely to be due to a smaller contribution to the natural remanence from multidomain magnetite.

The average recoverable low-temperature demagnetization is 45 ± 9 per cent for natural remanence, which is larger than the 24 ± 3 per cent average for saturation remanence in these six samples. Large recoverable low-temperature demagnetization of natural remanence, averaging 57 ± 19 per cent, can also be inferred for the four Whin Sill dolerite samples bearing low-titanium magnetite studied by Creer & Like (1967), if one corrects for the large effect due to the presence of the Earth's field in their experiments. Such large recoverable demagnetization of natural remanence suggests a large contribution to natural remanence from pseudo-single-domain

magnetite (since low-coercivity multidomain magnetite should contribute mainly to permanent demagnetization, and elongated single-domain magnetite grains should contribute little to either recoverable or permanent demagnetization). The large recoverable low-temperature demagnetization of natural remanence may be due to the pseudo-single-domain magnetite possessing high internal stresses, which may also be responsible for the high coercivity of the natural remanence.

5 DISCUSSION OF DOLERITES DOMINATED BY SINGLE-DOMAIN MAGNETITE

For elongated single-domain magnetite particles of random orientation, theory (Stoner & Wohlfarth 1948) predicts

$$H_C = C(N_b - N_a)J_S, \quad (4)$$

$$J_{RS}/J_S = 1/2. \quad (5)$$

Here, N_b and N_a are the respective self-demagnetizing factors along and perpendicular to the long axes of the particles and $C = 479$ if H_C is expressed in millitesla ($C = 0.479$ in cgs).

Our three dolerite samples of highest H_C (≥ 40 mT) are thought to be dominated by elongated single-domain magnetite particles, because H_C shows little change on cooling to ~ 135 K (Fig. 3). This is expected from eq. (4), since N_b and N_a should remain constant and J_S should only increase by ~ 5 per cent on cooling to ~ 135 K (Pauthenet 1950). Room-temperature J_{RS}/J_S values range from 0.37 to 0.45, which is a little below the 0.50 expected from eq. (5), but this may partly be due to magnetostatic interaction between the magnetite particles in intergrown magnetite-ilmenite grains (Hodoch 1996).

Cooling our three dolerite samples of highest H_C to ~ 135 K causes relatively little change in saturation remanence (Fig. 3), as expected from eq. (5). Nor is there much change on cooling through the Verwey transition temperature T_V . (The small changes observed, particularly in dolerite 5901, are likely to be due to a small fraction of magnetite in pseudo-single-domain particles.) Similar behaviour is reported by Halgedahl & Jarrard (1995) for Lambertville dolerite plagioclase, whose J_{RS} is likely to be carried by exsolved single-domain magnetite particles. (In contrast, their glass-ceramic sample containing $0.1 \mu\text{m}$ magnetite grains shows much larger low-temperature demagnetization, perhaps because the magnetite grains have little elongation.)

Our three dolerite samples of highest H_C show relatively little change in H_C on cooling (as shown by triangles joined by dashed lines in Fig. 3). The saturation remanence given at 77 K and when warmed in zero field shows little change at T_V (as shown by stars joined by dotted lines in Fig. 3). Again, the small changes observed, particularly in dolerite 5901, are likely to be due to a small fraction of pseudo-single-domain magnetite. This lack of change at T_V may be due to suppression of the Verwey transition by surface oxidation of the magnetite particles, as observed for synthetic magnetite grains by Özdemir *et al.* (1993). Assuming surface oxidation does not affect the magnetostriction of the magnetite above T_V , the absence of much change in saturation remanence on cooling to ~ 135 K supports dominance by elongated single-domain magnetite particles in our three dolerite samples of highest H_C (≥ 40 mT).

6 PALAEOMAGNETIC APPLICABILITY OF LOW-TEMPERATURE DEMAGNETIZATION

The permanent demagnetization caused by a cooling cycle to liquid-nitrogen temperature (77 K) is easy to measure and can be palaeomagnetically useful. It preferentially removes the low-coercivity remanence carried by multidomain magnetite from the higher-coercivity remanence carried by single-domain or pseudo-single-domain magnetite (Merrill 1970; Levi & Merrill 1978; Dunlop & Argyle 1991; Heider *et al.* 1992). For example, Dunlop, Özdemir & Schmidt (1997) find that a cooling cycle applied before thermal demagnetization of natural remanence in a monzonite removes the 'anomalously' high-unblocking-temperature thermoviscous component due to multidomain magnetite.

Halgedahl & Jarrard (1995) suggest that measuring remanence at room temperature after cooling cycles to lower and lower temperature will progressively remove the multidomain component of a natural remanence carried by magnetite, allowing it to be resolved accurately on a vector endpoint diagram. Our low-temperature cycles to 135 K and 77 K demonstrate progressive permanent demagnetization, supporting this suggestion.

We wish to point out that it may be palaeomagnetically useful to measure *recoverable* as well as permanent low-temperature demagnetization. As well as measuring remanence at room temperature before and after the cooling cycle, we suggest measuring the remanence direction and intensity while the sample is at 77 K (which should not be technically difficult in a cryogenic magnetometer). The remanence at 77 K should include all single-domain magnetite remanence. The remanence increase on warming back to room temperature should be mainly due to pseudo-single-domain magnetite, and its direction and intensity can be obtained by subtracting the remanence vector at 77 K from the remanence vector present on warming back to room temperature. Hence, it should be possible to separate the pseudo-single-domain from the single-domain remanence, which can be useful when the former rather than the latter is primary.

For example, Van Velzen & Zijdeveld (1990) studied Pliocene marine marls whose primary remanence is carried by pseudo-single-domain or single-domain magnetite and is overprinted by a secondary remanence of opposite polarity carried by smaller single-domain magnetite grains. Alternating-field demagnetization did not reveal the primary component, whereas thermal demagnetization did. Measuring the recoverable low-temperature demagnetization might also reveal the primary component carried by pseudo-single-domain magnetite, without the changes in magnetic properties that thermal demagnetization often causes.

However, note that permanent low-temperature demagnetization usually does not completely remove the remanence carried by large multidomain grains (as can be seen in Fig. 5b), nor does measuring recoverable low-temperature demagnetization completely separate pseudo-single-domain from large-multidomain-grain remanence (since the latter shows small recoverable demagnetization, as seen in Fig. 5b). Also, titanium content or surface oxidation of magnetite grains may suppress the Verwey transition (Özdemir *et al.* 1993), reducing the amount of low-temperature demagnetization and complicating its interpretation. Hence, we do not expect low-temperature demagnetization ever to be as widely

applicable to palaeomagnetism as alternating-field or thermal demagnetization. Nevertheless, permanent low-temperature demagnetization is a simple and often useful pre-treatment for thermal demagnetization of magnetite-bearing rocks (Schmidt 1993). Measuring recoverable low-temperature demagnetization is not as technically simple, but should be useful in isolating the natural remanence component carried by pseudo-single-domain magnetite.

7 CONCLUSIONS

The low-temperature demagnetization of saturation remanence is compared for samples containing multidomain, pseudo-single-domain and single-domain magnetite in Fig. 6.

Multidomain magnetite shows a large decrease in saturation remanence on cooling to 77 K in zero field, as has long been known (Ozima *et al.* 1967). Most of this demagnetization is permanent, although a small part may recover on warming back to room temperature in zero field. On average, for multidomain crushed natural magnetite of grain size 210–250 μm (this paper), 100–150 μm and 30–40 μm (Hartstra 1982), the saturation remanence shows a permanent demagnetization of 76 ± 5 per cent and a recoverable demagnetization of 11 ± 5 per cent on cooling to 77 K. Most of the permanent

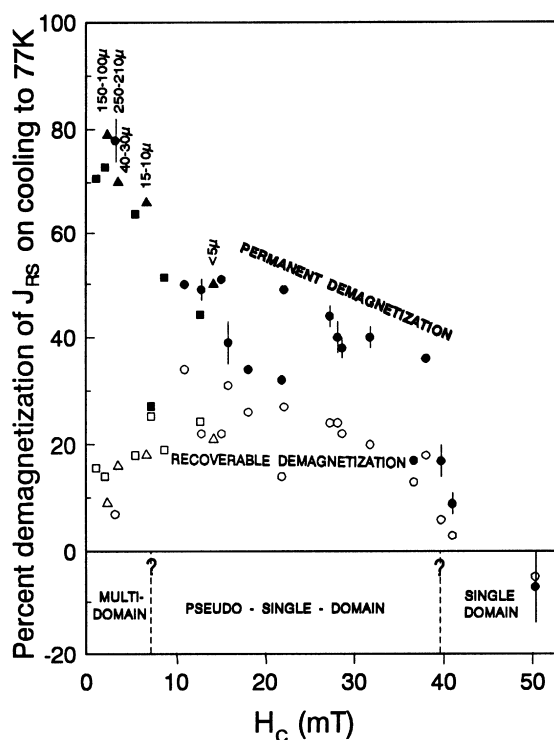


Figure 6. Solid symbols show the percentage permanent demagnetization of saturation remanence (J_{RS}) on cooling to 77 K in zero field plotted against coercive force (H_C). Open symbols show the same for recoverable demagnetization (saturation remanence decrease at 77 K that is recovered on warming back to room temperature in zero field). Circles are for our dolerite samples and the 210–250 μm crushed magnetite sample (with error bars shown for the permanent demagnetization). Squares are for the mafic igneous rocks of Hodych (1991). Triangles are for samples of Hartstra (1982), with grain sizes of crushed magnetite indicated. Ranges of H_C in which the magnetite is likely to be multidomain, pseudo-single-domain or single-domain are shown.

demagnetization occurs while cooling to ~ 135 K (the 'isotropic' point of magnetite) and little is associated with the Verwey transition (at ~ 122 K). The decrease on cooling to ~ 135 K occurs more rapidly for the saturation remanence than for H_C or \bar{J}_S/J_S . Perhaps a decrease in \bar{J}_S/J_S lowers stress-induced barriers to domain-wall motion and affects the remanence, not only by lowering coercivity, but also by promoting domain reorganization.

In our 13 dolerite samples whose magnetite is likely to be pseudo-single-domain ($H_C < 40$ mT, $J_{RS}/J_S < 0.35$), cooling to 77 K in zero field causes a permanent demagnetization of saturation remanence averaging 40 ± 9 per cent (about half that in multidomain magnetite) and a recoverable demagnetization averaging 23 ± 6 per cent (about twice that in multidomain magnetite). The remanence decrease on cooling to ~ 135 K (averaging 37 ± 10 per cent) is mostly permanent and often occurs in rough proportion to H_C and \bar{J}_S/J_S , as expected if the coercivity is due to stress-induced barriers to domain wall motion. The additional remanence decrease on cooling from ~ 135 K to 77 K (averaging 26 ± 9 per cent) seems mostly to be due to the Verwey transition forcing domain reorganization. The increase in remanence on warming back to room temperature (averaging 23 ± 6 per cent) may be due to internal stresses acting as a bridge between different easy axes below the Verwey transition and above the isotropic point [as suggested by Kobayashi & Fuller (1968) and by Heider *et al.* (1992)]. This recoverable low-temperature demagnetization is even larger for natural remanence, averaging 46 ± 9 per cent for the six dolerites measured. We point out that large recoverable low-temperature demagnetization seems characteristic of pseudo-single-domain magnetite with high internal stresses in our dolerite samples and that the remanence component due to such magnetite may be isolated by measuring recoverable low-temperature demagnetization.

Only our three dolerite samples of highest coercive force ($H_C \geq 40$ mT, $J_{RS}/J_S > 0.35$) show little change in H_C and saturation remanence on cooling, suggesting that their remanence stability is due to shape anisotropy in single-domain magnetite grains. In most of our dolerite samples, the saturation remanence stability seems to be due to pseudo-single-domain magnetite in which domain wall motion is opposed by internal stresses. This may also be true for the natural remanence, since the average alternating field required to demagnetize the remanence by half is only 25 per cent lower for the saturation remanence than for the natural remanence. The internal stresses are likely to be due to the magnetite being intergrown with fine ilmenite lamellae, as is common in mafic igneous rocks. Hence, we expect that internal stresses in pseudo-single-domain magnetite are often important in preserving the palaeomagnetic record in mafic igneous rocks.

ACKNOWLEDGMENTS

This research was supported by a grant from the Natural Sciences and Engineering Research Council of Canada. B. Ryan of the Department of Natural Resources of the Government of Newfoundland and Labrador and K. L. Buchan of the Geological Survey of Canada are thanked for help in obtaining the dolerite samples. We thank A. J. Gollop, D. C. Sheppard, S. C. Hodych, P. J. A. McCausland and R. Pätzold for help with the low-temperature demagnetization experiments. R. Pätzold is also thanked for preparing the diagrams.

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